

## ELEVATED TEMPERATURE STRAIN GAGES\*

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The Host Program has as one of its goals the development of electrical resistance strain gages for static strain measurements at temperatures  $\geq 1273^{\circ}\text{K}$ . In order to attain this goal strain gage materials must have a characteristic response to strain, temperature and time that is reproducible or that varies in a predictable manner within specified limits. The principal contributions to the relative change in resistance are (1) the change in resistance with strain, (2) the change in resistance with temperature (coefficient of thermal resistance) and (3) time dependent changes in resistance due to chemical changes (e.g., oxidation) and dimensional changes (creep) (ref. 1). These then are the principal parameters to be considered in the selection of materials for strain gage elements. However, in order to proceed with the development of such gages the selection of material for the gage elements, consideration must be given to material parameters that effect electrical resistance. The material factors are: phase, magnetic and order-disorder transitions, short range ordering, clustering, plastic deformation and chemical changes associated with material reactions with an elevated temperature environment. These material parameters are interrelated and their effects on elevated temperature electrical resistance are both temperature and time dependent (refs. 2 and 3).

Prior to the selection of a list of materials for potential strain gage applications, we set out to establish the criteria to be used in the selection of material for evaluation. Phase diagrams of binary alloys were utilized as the first step in the selection of materials that have sufficiently high melting temperature, do not undergo a phase transformation or a magnetic transition. Since phase diagrams usually provide no information on structural changes associated with order-disorder transitions or clustering, we made use of thermodynamic data when available to select ideal solid solution alloys. Alloys that are ideal solutions should not undergo transitions such as clustering, order-disorder, etc. The basis for classifying an alloy as an ideal solution is that the activity coefficient is equal to one. Alloy compositions with activity coefficients of the two components within about 15% of one were selected, see Table I. Several qualitative criteria have evolved to aid in the selection process when quantitative activity data did not exist. These qualitative rules suggest that alloys tend to form ideal solutions when the molar volume of the two components are equal and when the electronegativity of the two components are similar (refs. 4 and 5). In addition several alloys that have a linear electrical resistivity-temperature relation were included in the initial list of materials for evaluation (ref. 5). The list of binary alloys for the first phase of the investigation is listed in Table I.

In addition to the metallic alloys, evaluation of a series of transition metal carbides, nitrides and silicides, Table I, is also in progress. These materials were selected based upon the following: (1) high melting temperature, (2) good mechanical strength at elevated temperature, (3) low and metallic like resistivities, (4) the absence of phase transitions within the  $0^{\circ} - 1000^{\circ}\text{C}$  temperature range,

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(5) good oxidation resistance and (6) thin films can be etched for pattern geometry (ref. 6). In addition to the six compounds (TiC, TiN, ZrC, ZrN, TaSi<sub>2</sub> and TiSi<sub>2</sub>) selected as most promising for evaluation, interest in SiC has evolved. This semiconducting compound is of interest because of its stable physical characteristics, similar to those described above for the transition metal compounds, at temperatures up to 1200°C, as well as the observation that efforts to develop SiC elevated temperature gages are in progress in Russia (ref. 7).

#### MATERIAL PREPARATION AND PROCESSING

The alloys listed in Table I are being prepared by arc melting in the usual manner. The homogenized ingots were processed into specimens by electric discharge machining or slicing with a diamond wheel. Specimens of CrV were 25 x 2.1 x .406 mm, Al-V 12 x 1.8 x 1.5 mm. Specimens have also been prepared by means of drawing the molten alloy into a quartz tube connected to a vacuum. This latter technique produced longer wire specimens. So far our efforts to produce specimens of MoRe and MoW by the quartz tube technique have not been successful.

Samples of SiC, TiN, TiC in thin film form have been obtained from several sources. These samples were prepared by sputtering or by CVD techniques. Specimens of SiC on a Al<sub>2</sub>O<sub>3</sub> substrate were cut to 13 x 1.2 mm and 2500 Å thick.

The electrical resistance of the specimens was measured via a four probe technique using Pt leads and pressure contacts. The contacts were checked and found to be ohmic at all temperatures. Measurements were conducted in a quartz evacuated tube that contained an Al<sub>2</sub>O<sub>3</sub> specimen holder with a metal tube envelope surrounding the quartz tube. The vacuum was better than 10<sup>-5</sup> Torr and the temperature gradient was found to vary by about 2 - 10°C along the specimen holder, with a temperature variation over the length of the specimen of ~ 2-4°C at the elevated temperatures.

#### RESULTS

The resistance-temperature data was obtained with a heating rate of ~ 1.25°C/min. and a cooling rate of ~ 0.6°C/min. The resistance data is an average of approximately 8 measurements that were made after a sojourn of about 30 minutes at the temperature of interest. Thermal EMF's were eliminated by changing the current direction. Figure 1 is a plot of resistance vs temperature for a nominal 69.1 w/o (70 a/o) V-Cr alloy. The resistance varied linearly with temperature, with a slope of ~ 2.68 x 10<sup>-3</sup> mΩ/C, there is good agreement for the two cycles shown, furthermore the drift at 1000°C after 15 hrs. was relatively small. Figure 2 shows the resistance drift as a function of time, the drift after 900 minutes was ~1.7 %. Table II relates the resistance drift for short times at intermediate temperatures. From figure 1, the thermal coefficient of resistance at 1000°C was found to be ~ 5.6 x 10<sup>-4</sup>/°C.

The resistance-temperature data for the 81.6 w/o (70 a/o) V-Al alloy is shown in figure 3. After the first heating cycle, the resistance varied reproducibly with temperature. The origin of the initial high resistance from room temperature to ~ 500°C is unknown. The decrease in resistance with temperature suggests that we repeat these measurements to insure that they are not due to a fault in the measuring technique. If repeated measurements produce the same temperature dependence, that would indicate semiconducting electronic behavior.

Figure 4 shows the resistance-temperature data for SiC sputtered film on an  $\text{Al}_2\text{O}_3$  substrate. The specimen was initially conditioned by heating and cooling from  $1000^\circ\text{C}$ . The resistance drift at  $1000^\circ\text{C}$  during the first heating cycle was associated with a 17 hr. sojourn at  $1000^\circ\text{C}$ . After the first heating cycle, there is some indication that the resistance-temperature relation is constant. However, as indicated in figure 5, the resistance showed a positive drift with time at 840 and  $900^\circ\text{C}$ , while a negative drift was observed at 949 and  $1000^\circ\text{C}$ . Inasmuch as we observed extensive contact reactions between the Pt wire and SiC, the present data is suspect and we are in the process of examining other probe materials.

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TABLE I  
MATERIALS SELECTED FOR EVALUATION

Material	Basis for Selection
Pd-Ag	1
Al-V	1
Nb-V	2
Mo-W	2
Hf-Zr	2
Pd-Rh	2
Cr-V	3
Ta-W	3
Mo-Re	3
TiC	4
TiN	4
TiSi <sub>2</sub>	4
ZrC	4
ZrN	4
TaSi <sub>2</sub>	4
SiC	4

1. Ideal solution, thermodynamic data.
2. Qualitative parameters, tendency to form ideal solutions.
3. Resistance-temperature data.
4. Refractory compounds, high melting temperature, structural stability, oxidation resistance.

TABLE II  
RESISTANCE DRIFT FOR CrV ALLOY, m $\Omega$

TEMPERATURE (°C)	TIME	0	1/2 h	1 h
429.9		3.376		
428.78			3.392	3.283
581.75		3.749		3.755
586			3.743	
703		4.179		4.081
705.5			4.084	
845		4.481		4.486
850			4.484	

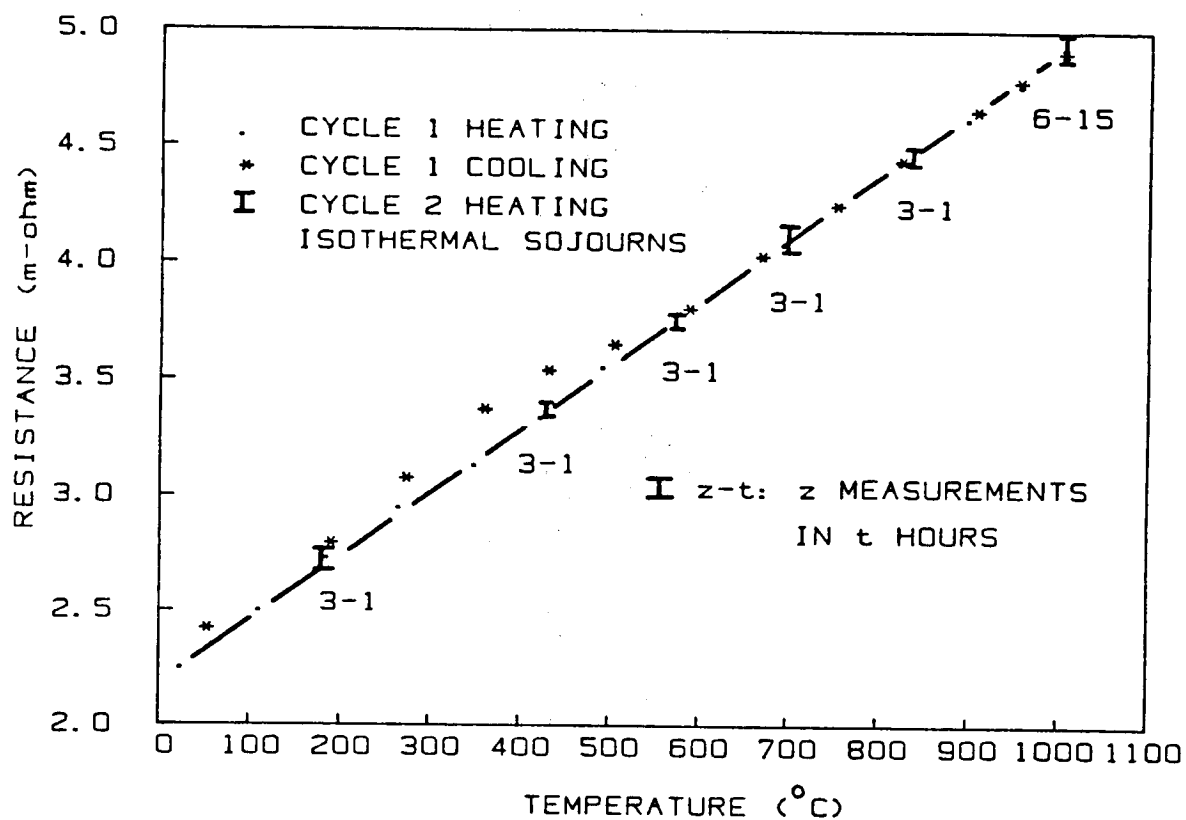


FIGURE 1 RESISTANCE Vs. TEMPERATURE OF Cr-V

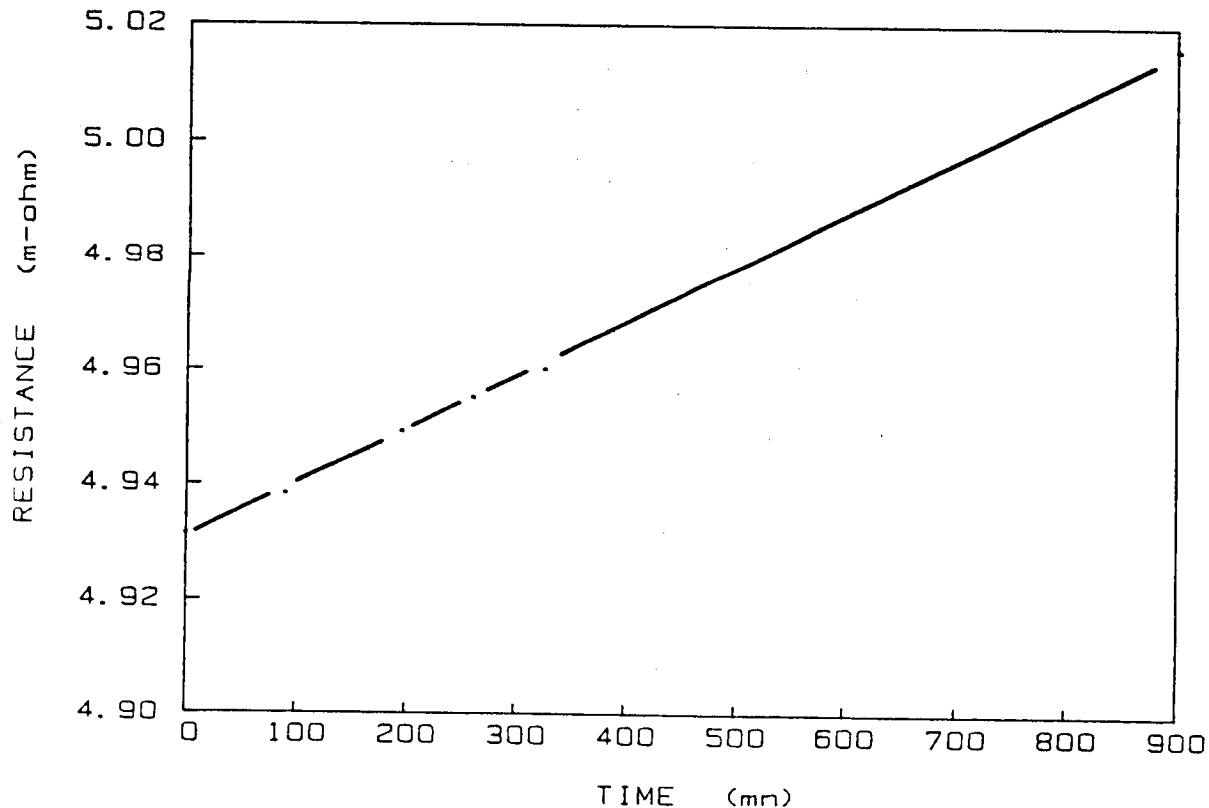


FIGURE 2 RESISTANCE DRIFT Vs. TIME at 1002.5°C OF Cr-V

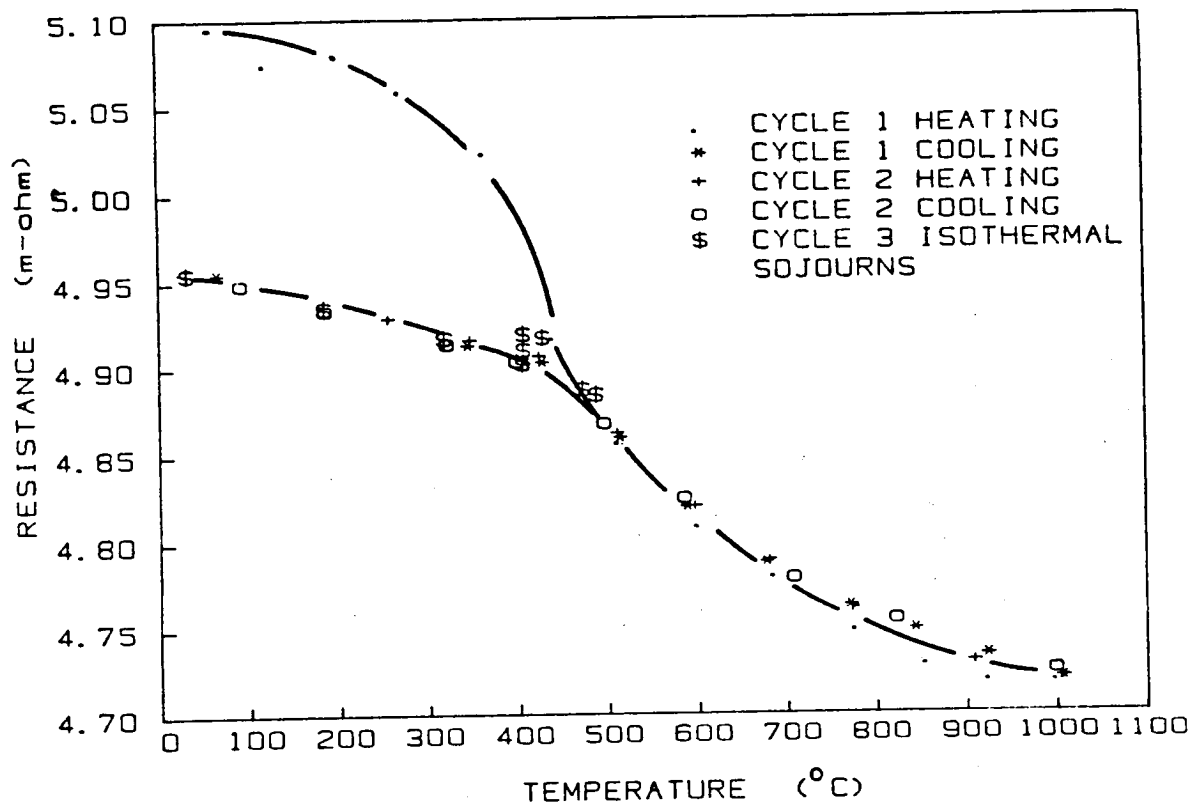


FIGURE 3 RESISTANCE VS. TEMPERATURE OF AlV

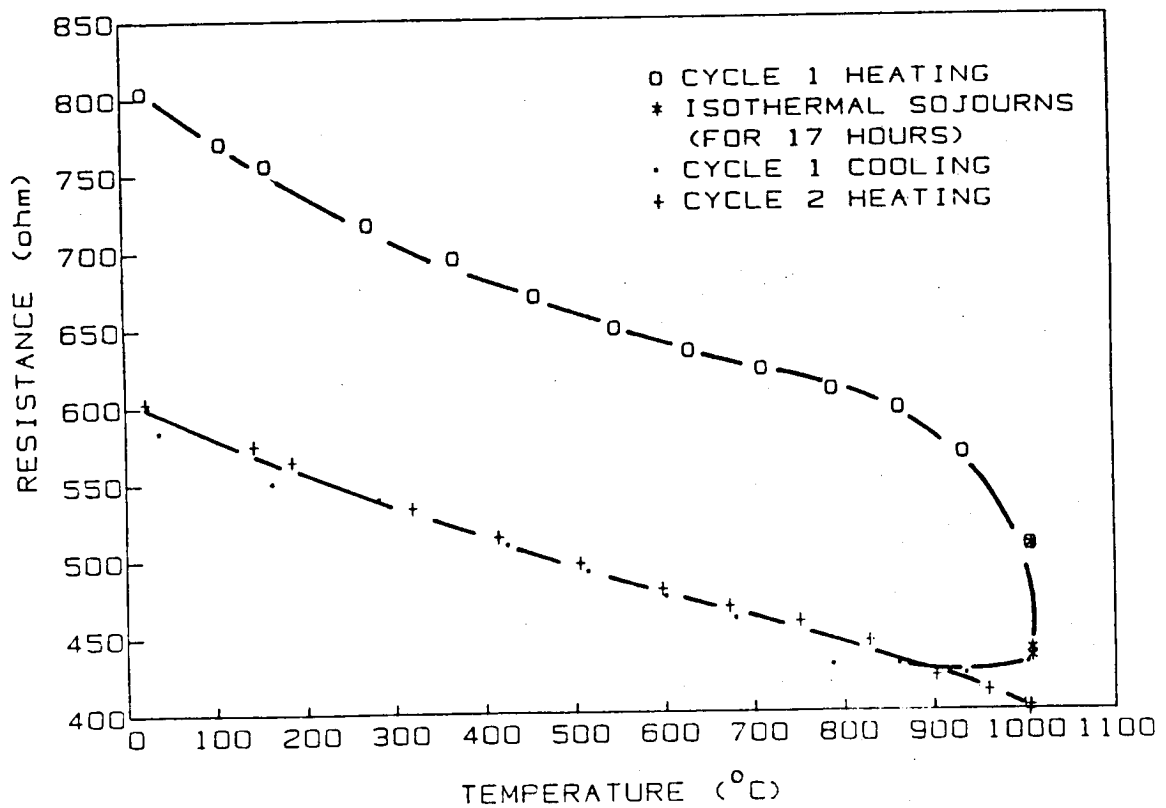


FIGURE 4 RESISTANCE VS. TEMPERATURE OF SiC

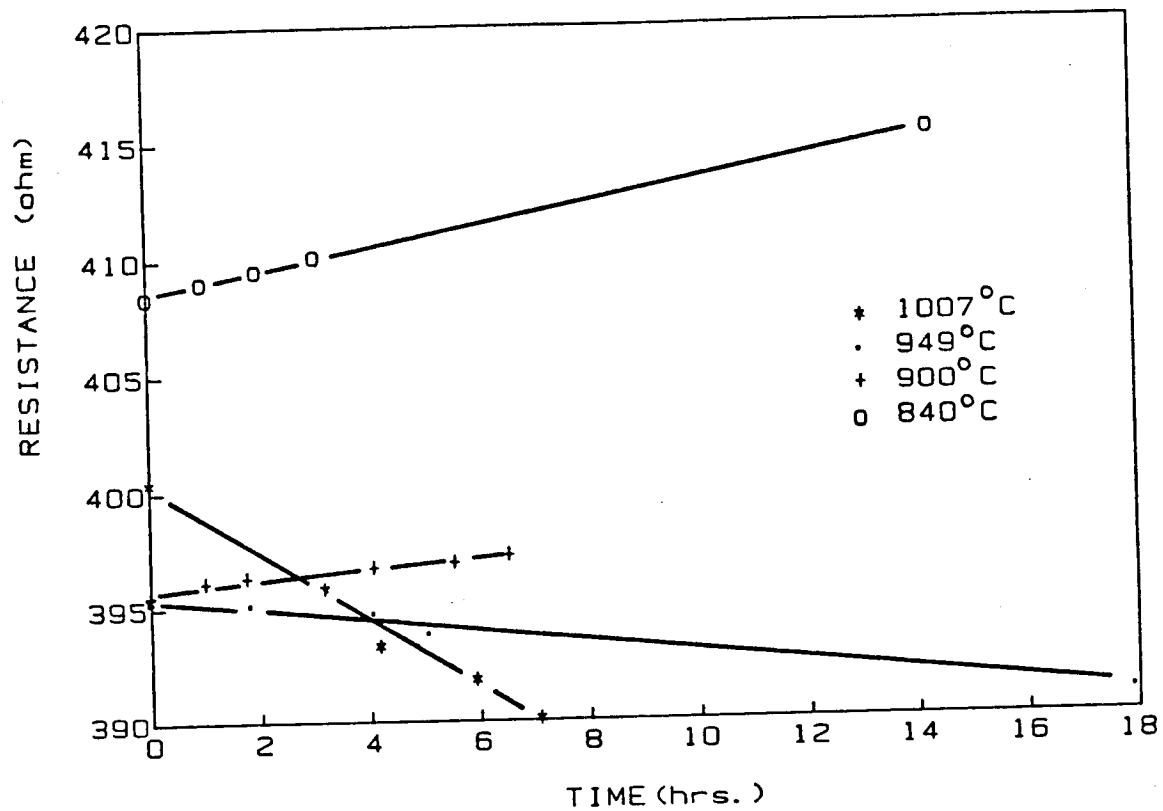


FIGURE 5 RESISTANCE DRIFT Vs. TIME OF SiC